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(54) AN ELECTROLYTE PURIFICATION METHOD AND A REFINER TO APPLY THIS METHOD (VARIANTS)

(57) The invention is related to preparation of ultra-pure electrolytes.

The goal of the invention - to improve the quality, efficiency, and commercial attraction of electrolyte purification, the refiner compactness.

The goal is achieved because the method of purification with low-voltage electrophoresis and sedimentation is sumplemented with superadditive high-current electrophoresis procedures, heating and crystallization, i.e. the refiner comprising a sludge pretreatment unit 1 was equipped with a series-connected filtration unit 2, a control unit 3 and a crystallizer 40 connected to an input of the treatment unit, and via the second input - to the output of the filtration unit, data outputs and control inputs of units are connected to the inputs and outputs of the control unit. The operations added to the method, their interactions, and the units added to the refiner, their components and connections are used for the first time.

The method and the refiner can be used also in the production of coatings and foils of high-pure metals, healing water and medicine.

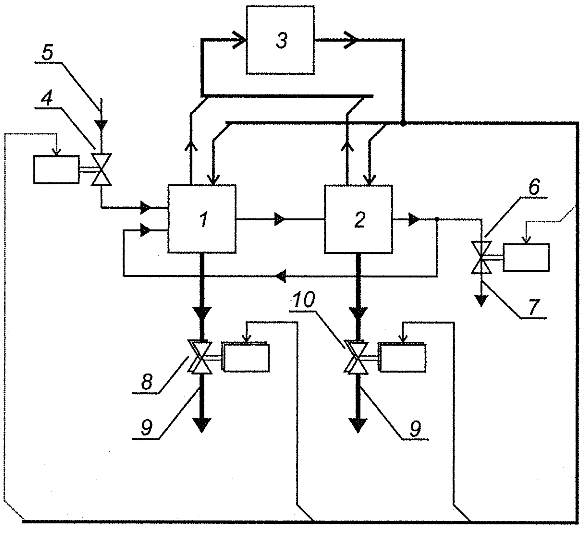

Fig. 1.

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Description

[0001] The invention is related to electroplating, namely to methods and devices for continuous renovation, removal of insoluble particles and *ultra-pure* electrolyte obtaining. Currently for electronic and electrotechnical industries' needs, a lot of superpurity metal is required to obtain previously unreachable parameters of devices manufactured with layered ferromagnets and layered electrical conductors of these metals. Thus electroplating of superpurity metals from their aqueous salt solutions are considered as the cheapest and most commercially *viable.* The quality of metal deposited chemically or electrochemically on solid or liquid surface of its aqueous salt solution depends primarily on the chemical purity of this solution [1]. Just like in the water, the main type of contaminanation, impossible or economically unreasonable to be removed from solutions by filling-in chemical reagents, if the required solution chemical composition is observed, in most cases is suspended insoluble fine particles within 1 nm ... 100 nm [2]. Most of these fragile sludge particles are inevitably involved into the deposited metal structure, taking an electric charge during chemical and electrochemical deposited metal. Because of sludge fineness in comparison with the distance between ions of the crystalline lattice of the compact metal less than 0.5 nm, even the weight concentration of 10^{-4} % ... 10^{-2} % of the solution particles is sufficient to create mechanical stresses in the deposited metal which parameters may cause its cracks or result a dark powder layer instead of the desired ductile metal one. Therefore, the use of impure electrolyte, to reduce their cost, results in failure to obtain the proper quality of metal coating. Insoluble sludge particles of the same undesirable property may occur in addition to organic and inorganic colloids in the water as insoluble particles of simple and complex compounds of silicon, aluminum and other chemical elements and their solvates, lyophilic (with high wettability by the liquid phase of the solution), due to the high nucleus charge in these particles, and loose, often having an indistinguishable density compared to contaminated solution, therefore, not nonremovable by sedimentation, centrifugation, filtration and distillation in salt solutions of used metal with components of these salts. Solutions of transition metal salts, changing its valency during air oxidation of these salts with insoluble cementite particles [1], as well as during the process involving these solutions [3] are particularly susceptible to this contamination method. It should be noted that highlypure solutions allow to obtain very-high-purity iron and other very-high-purity transition metals not only by electroplating, but also by dense chemical deposition restoring these metals with hydrazine or other reducing agents (pg. 366 in [4], pg. 201 in [5]. Therefore, to obtain very-high-purity salt solutions with removed colloidal sludge is considered to be relevant.

[0002] An analysis of the first six analogues leads to the conclusion that in order to increase the electrolyte purification level and the purifying equipment efficiency, it is necessary to use the preliminary sludge coarsening process in the electrolyte before final electrolyte purification, by simultaneous treatments, each enhances the others' action. This is so-called conjugation (mutual acceleration) of two simultaneous impacts on a single substance, which at the same time significantly help each other, enhance each other's effectiveness. Such interaction at the electrolyte pretreatment phase can be superadditive when an effect of these processes is significantly superior to their total action, namely improves the sludge purification efficiency by 2 ... 10 times and improves its quality by 10 ... 1000 times.

[0003] Thus the methods of sludge treatment by adding new components into the electrolyte, causing coagulation and flocculation, but contaminating the electrolyte, should be refused. In (part 4 «Colloid chemistry» in [6]) and (p. 162 in [7]), except strong dilution or concentration, which are not acceptable due to changes in electrolyte parameters, also external physical impact is considered which causes and stimulates coagulation and flocculation as lyophobic (poor wettable) and lyophilic (high wettable) sludge particles. These effects include ultrasonication, electric field exposure, heating and freezing of electrolyte. Coarsened particles can be removed fully and high-efficiently from an electrolyte by sedimentation, flotation or filtration.

1. A device for automatic continuous liquid filtration is known [8], which comprises a vacuum filter consisting of an upper chamber and a filtrate chamber separated by a mesh partition with filter elements, a liquid feeding pipe to the upper chamber with a float valve, a compressed air line, control valves, a cylindrical rising pipe connected at its top via conduit to a passive ejector nozzle and a vacuum filter spill pipe, and installed at the bottom into an open container filled with filtered fluid. Obviously, the used mechanical filtration is considered not only as more productive method of final fluid purification, as compared to the slow purification method known for a long - sedimentation, but also one of the fastest. High performance of the device is also provided by integrated improvement, i.e. pressure difference at the sides of the mesh partition with filter elements. The disadvantages of the device are: periodic stops to replace and clean filter elements, clogged up with fine sludge, in spite of its dissociation; the impossibility to clean fluid from fine particles of 1 ... 10 nm, since fluid pretreatment ensuring mutual adhesion (coagulation) of these particles and their subsequent enlargement (flocculation) are not applied in the device, however it could help reach complete dissociation of sludge from liquid by final purification, i.e. filtration.

2. A device for liquids cleaning is known [9], which comprises a flotation chamber with liquid inlet and

outlet nozzles, outlets of gas, foam, and sludge, a foam receiver, and means for vacuum, formed as a vertical container with discharge nozzles of the clarified liquid, gas, sludge, a foam discharge nozzle with a foam riser and a horizontal baffle, with vertical tubes, wherein the fluid outlet of the flotation chamber is connected to the vertical reservoir via the pipeline, an outlet is located above the baffle, a gas discharge nozzle of the flotation chamber is connected to the container at the top and a foam outlet is located above the lower edge of the vertical pipes. The device removes sludge particles by flotation. The disadvantages of this method include incomplete fluid purification from sludge particles, especially in their lyophilic (high wettability), expenditures of the dissolved salt in foam formation, salt film formation during concentrated solution evaporation, the impossibility to use the device for removal of transition metal salts solutions due to their rapid oxidation and transfering into sludge during air bubbling of the solution.

3. The method of purification of plating bath electrolyte is known as well as a vertical centrifuge for this method [10], thus the method includes centrifuging of initial electrolyte and the centrifuge consists of a cylindrical case with a cover and a stop mounted on the upper and lower thrust roller bearings, nozzles and chemically resistant plastic seal glands, mounted in the cover and the stop of the body, and a centrifugal pump with a hose and a fixed drain funnel connected to the upper pipe. Comparing to sedimentation, the centrifugation method is a high-efficient method of final removal of sludge from electrolyte. At centrifugation, sludge coagulation is similarily observed, which is caused by increased concentration, i.e, the process is better apply before the final removal of sludge from the liquid. However, in the used method as well as in other methods of final removal of fine suspended particles from fluid, i.e. sedimentation and filtration, this process operates only in the sediment and does not improve the quality of purification. The disadvantage of this method is its ineffective use for removal of sludge as loose lyophilic particles with homogeneity, i.e. of almost the same density as the liquid phase of the solution of their settlement. Thus the size of the suspended particles can be observed to be substantially higher than 1 ... 10 nm, and of their flakes often reaches 5mm or more. The device is not automatic, so another disadvantage is expenditures associated with regular stops and discharges of accumulated sludge from the centrifuge case.

4. A method of fluid purification by microfiltration and ultrafiltration is known [11], [12], [13], including fluid filtration from particles of sizes from 10nm, through semipermeable membranes under pressure of about 8 ... 10 bar. Despite the method also does not provide for other fluid purification, being the single and finishing treatment method, it provides *ultra-pure* electrolyte of acceptable quality. Its disadvantage is the low performance at optimum dimension, or large and complex design at optimum performance. Structures implementing the method may be suitable for purification only of limited liquid volume, since they are quickly clogged and become inoperative without complete replacement of chemically resistant semipermeable membranes. The high costs associated with membrane replacement also lead to commercial unattraction of electrolyte purification method.

5. The natural and municipal wastewater filtration method is known [14] as well as water clarification methods (see pg. 63 [15]), including their treatment with coagulants (chloride, poly-diallyl-dimethyl-ammonium) and flocculants (lime) before final purification by sedimentation and mechanical filtration. The treatment with chemical coagulant and flocculant seems to provide fast coarsening of sludge particles from 1 nm to 100 nm (coagulation and flocculation), therefore, high-speed quality final removal of these particles from water by sedimentation and/or filtration using low-cost and clogging-free mechanical filters. The disadvantage of the methods is the impossibility to apply it for electrolyte purification in the form of aqueous salt solutions of iron and other transition metals because of chemical reaction with dissolved salts of metal and salt capture from the solution into the sludge during coagulation. Thus coagulation of all suspended fine particles, differing in electric charge due to the different chemical compound, by the used methods of chemical bonding and salting-out is impossible. The disadvantages of this method are also constant consumtion of coagulants and flocculants and the need to remove their hard-to-remove residues from the electrolyte, both inorganic and organic, which contaminate the electrolyte and will be involved into the deposited metal. Consequently, the activation method for removal of undissolved fine sludge particles by adding coagulants and flocculants not only increases cost and complicates the purification process but considered as unacceptable for electrolyte purification used for chemical and electroplating of iron and iron group metals.

6. The method of purification and regeneration of the concentrated spent chromium-containing solution is known [16], including the solution heating, an alkali agent addition to reach pH = 5 ... 7, dissociation of the sediment by filtration, and the solution correction according to pH by adding chromium trioxide in an amount equal to the added alkaline agent, thus the solution heating is carried out up to 60 ... 80 °C, and flocculant - water soluble polyelectrolyte - is added before an alkaline agent. The method includes two

methods of electrolyte pretreatment, the first one - chemical coagulation and flocullation, and the second one - coagulation and flocullation caused by heating, which act simultaneously, increasing the efficiency of each other, i.e. are superadditive. At the same time the method also provides restoration of the main electrolyte parameters: its *pH* value and concentration of the selected salt of working metal. The disadvantage of this method is also the necessity of removal of residues of coagulants and flocculants from electrolyte.

7. The device for removal of the sludge from electrolyte is known [17], which comprises a supporting frame with the drive shaft system mounted, consisting of a corset-shaped driveshaft, a cylindrical countershaft, barrel and pinch shafts with an endless elastomer-contoured filter belt with driveshaft throats, passed between, a two-section tank, two ultrasound emitters, thus the lower filter belt branch above the first section of the tank is resiliently formed as barrel-shaped shafts in the form of an inclined chute, above which a liquid drain collector and an ultrasonic source immersed in the purified liquid are located, and a brush drum, an auger conveyor and the second ultrasonic source, immersed into the washing liquid, are installed in the second tank section. The device is not equipped with follow-up systems ensuring the ultrasound resonance in the liquid irrespective of flowrate fluctuations, viscosity changes at temperature changes, including affected by cavitation, as well as changes in its total concentration during sludge coagulation and dispersion that could provide coagulation in the sludge liquophobic nodes of standing ultrasonic waves. Obviously, an opposite action of ultrasound is observed in antinodes of waves, i.e. sludge dispersion (see pg. 251 [2]), which results in an equal liquid concentration ratio to the coarse and milling sludge. Therefore, the ultrasound effect relating to the other simulataneously used methods of sludge coarsening is antagonistic, not increasing but destroying the results achieved by all other simultaneously applied methods of sludge coagulation. Another disadvantage of the device is that during the fluid selection from ultrasonic waves nodes, the liquid with fine sludge will be inevitably selected from the rest of its volume. The device does not allow to obtain electrolyte of acceptable high-purity by full removal of coarse sludge by high-performance filtration.

8. The device for suspension clarification is known [18], which comprises a vertical cylindrical cone, a coaxially mounted inverted cone with a sediment discharge pipe, an annular reflector and nozzles for initial suspension input and purified liquid output, a vibrator connected to the case by a drive, wherein the case bottom is formed as an inverse cone with an inlet for initial suspension at the upper part, and the device is equipped with a conical divider placed above the initial suspension inlet pipe. Thanks to reflected suspension flows, the sludge particles actively collide with each other and stick together, i.e. coagulate, and in opposite motion of falling flakes of the compacted sludge and of the liquid impured of the sludge with the small sludge particles, these small particles collide and couple with falling flakes, even more compacting them, which form new dense flakes of sludge colliding due to the further cohesion, i.e. flocculate. The collision and adhesion of large and small sludge particles and their sedimentation result in lowering sludge concentration in the upper portion of the liquid flow, therefore, clarification of effluent liquid is provided. However, the collision of lyophilic, i.e. well wettable, particles of 1 ... 100 nm, tended to Brownian motion and homogeneous with respect to emulsion, substantially weakens by liquid, coagulation becomes insufficient, and these particles cannot be removed in the device. Only one type of electrolyte pretreatment is used in the device before sludge sedimentation - coagulation by particle collisions. Other treatments are not applied, especially those with mutual impact on the sludge. The main disadvantage of the device is associated - the inability to remove loose sludge particles unacceptable in the solution for metal deposition or in the electrolyte because it does not allow superpurity metal coatings.

9. The electric purifier of dielectric fluids is known [19], which comprises a case, purifying liquid inlet and purified liquid outlet nozzles and a filter element, thus the filter element is in the form of parallel conductors wound into a toroidal skein. Increasing in the electric field near the electrode conductors provides deposition on them, sludge concentration and electrocoagulation, then sedimented in the case bottom. The device is also known to dissociation of dispersed phase particles from the dispersion medium [20], which comprises a case with perforated collecting electrodes installed inside, a center electrode and a collection header, and the collecting electrodes are made hollow and connected to the collection header. Inhomogeneous alternating electric field with increasing stress near the collecting electrodes causes coagulation and further sedimentation of sludge particles. Electrocoagulation does not require chemicals, rising the technology in the cost and requiring additional processes to compensate contamination caused by these chemicals and electrolyte derating. The disadvantages of the devices are insufficient performance associated with the lack of interaction between the liquid volume with the electrode surface, and complexity of the quality control of the electrolyte.

10. The multiphase inductive electrocoagulator is known [21], which comprises a case with a conical bottom and a cover, and a current lead, made in the form of toroidal inductors with primary single-phase windings of high impedance bare wires, placed uniformly in the electrocoagulator working volume and connected to the primary polyphase network. The cost-effective economical heating of electrolyte is provided due to the heating of primary high impedance windings placed right in the electrolyte and secondary currents induced therein. Thanks to this heating, sludge particles are coarsened by sticking, i.e. coagulation and their further coarsening, i.e. flocculation, which provides sludge deposition by settling, i.e. sludge sedimentation. The disadvantages of the method applied in the electrocoagulator is the use of only one type of electrolyte pretreatment - sludge coagulation by heating and the use of low-productive method of final liquid purification - sedimentation. Thus the most undesirable sludge particles of 1 ... 10 nm, and therefore not subject to coagulation by heating, remain in the electrolyte. The structural disadvantage of the electrocoagulator is that the heating bare corrosion-resistant winding are covered with a thick layer of insulating dielectric sludge, overheating the windings and reducing the efficiency of heat transfer into the electrolyte.

11. The device for water purification is known [22], which comprises a thermostatic heat exchange capacity, means for influent water feeding for purification and means for drain of defrost water and liquid concentrate of impurities, means for water cooling and freezing, and means for ice melting with cooling and heating elements, the unit control associated with means for influent water feeding for purification and drain of melt water and liquid concentrate of impurities from the heat transfer capacity and connected to means for water cooling and freezing and ice melting, thus the heat exchange capacity is provided with a flat fissure inner cavity or an annular fissure cavity, and one of the walls of the heat exchange capacity, cooling and heating element free, made of transparent material and has one or more internal air cavities. Since the insoluble contamination particles, contained in the incoming water and not deposited due to their small size from 1 nm to 100 nm, during water cooling serve as the crystallization centers, dissociation and adhesion of these small crystals from water at the initial stage of its freezing facilitate concentration of contamination particles. Therefore, further water freezing forced out contamination from ice. At ice thawing, contamination particles are concentrated in the small local volume of salty water, coagulated actively due to their increased concentration and water salinity, and then flocculate, sedimenting and collecting as a film at the top of the container. This method of contamination displacement from the water does not require spending of coagulants and other consumables. A computer control unit with a water temperature sensor provides the device automatic operation with mode switching: filling water, freezing, thawing, separated drain of melt and dirty water. Separate drainage of contaminated and purified water require no mechanical filter, consequently no costs for filter replacement and maintenance are required. Two kinds of sludge pretreatment in water are used in the device, which, due to their actions sequence, do not have the ability to enhance each other's impact - concentration and coagulation, and during concentration, the sludge migration into the purified water and slow independent types of final purification - sedimentation and flotation. Therefore, the disadvantage of the device is the impossibility of its application for acceptable removal from the fluid of the contaminating fine sludge, involved in the ice because the sludge particles act as crystallization centers.

12. The filter for liquid purification is known [23], which comprises a thermally insulated cylindrical dielectric body, with a dielectric rod axially installed, electrodes of opposite polarity with an angle of 7 ... 10 ° to the body axis, thus the body and the rod are provided with ducts for the coolant, the rod is provided with hollow blades set at angle of 7 ... 10 ° to the body axis, and inlet and outlet conduits to the rod inside, and the inner surface of the body and the outer surface of the rod have spiral grooves. During fluid cooling, tiny crystals appear therein, mainly on the contaminated particles like on crystallization centers and of similar size 5 ... 100 nm, therefore coagulating them, linking to the electrodes and deposited on them. However, because of the low average speed of large crystals moving with colloidal particles during electrophoresis (see pg. 25 [6]) equal to v_{phores} = 1 ... 4 m/sec. Therefore, despite two simultaneous pretreatments of fine sludge particles are applied in the filter, i.e. coarsening of the particles by salt crystallization on them, and adhesion of small crystals by increasing their concentration near the electrodes, the impacts of these types of treatment are mutually weakened. Because of the small overlap of the cross-section of liquid flow and volume by electrodes, only 10 ... 15 % of crystallized contamination particles have time to deposite on the electrodes. These disadvantages of the filter do not allow its use for *ultra-pure* fluids and electrolytes.

13. A device for electrochemical treatment of drinking water is known [24], which comprises a vertical flotation chamber, an electrolyzer with a soluble electrode pack mounted directly under the flotation chamber, a filter and a final fine filter, thus the bottom of the flotation chamber is flat with a hole combined with an electrolyzer outlet, and equipped with a plate

located above this hole and overlapping it on the area, thus the polyester vertical filter baffle, spaced from the bottom of the flotation chamber at over 1/3 of its height, forms with the walls a closed cavity for purified water collection and discharge through the hole in its wall, another hole is situated in the cover of the flotation chamber for discharge of generated gases and sludge.

Due to the metal anode dissolution of the electrolyzer, water is saturated with ions, causing coagulation, i.e. coarsening by adhesion of fine particles from 1 nm to 100 nm, and therefore not deposited by decantation before the coagulation, and the particles by their chemical composition are mainly related to silicates, aluminates, hydroxides of heavy metals and other insoluble inorganic substances, PVA (polyvinyl-chloride), latex or other polymers, petroleum products, fats, oils and other products of organic synthesis. Simultaneously with anod metal dissolving, oxygen gas is mainly discharged on them, and hydrogen - in anode. Tiny bubbles of these gases provide flotation of coagulated contamination particles, i.e. they catch and carry them upward into the direction of treated water flow. Coagulation and subsequent formation of flakes, i.e. flocullation, end for the observed cross section of the water flow when the section reaches the lower edge of the filter.

The advantages of the device are simple design and the use of two conjugated processes of water pretreatment - electrophoresis and coagulation, which is provided by adding removable inorganic impurities by electrode dissolving. This treatment should improve the efficiency of sludge removal by filtration.

The main disadvantage of the device is frequent change of soluble anodes of electrolyzer located at the bottom of the device, and hence frequent removal from the water system. Since the electrophoresis effect is insignificant at an acceptable flow rate of filtered water, water pretreatment is presented only as sludge coagulation provided with metal ions from additional particulate sludge, and requires removal of this newly added contamination, and thus considered not highly efficient. Since the flotation sludge flush is partial, the filter is inevitably clogged, and since the electrode current and the water circulation rate are required to be decreased manually in accordance with the results of water contamination samples at the input and output of the device. Obviously, the acceptable quality of water purification fails to be provided as well as the consumption of electrodes and water discharged with the sludge to be minimized. The device can not be applied for metal salts solution purification due to the oxidizing effect of oxygen at the electrolyzer anodes.

14. The most similar technical solution is removal of suspended particles from solution [25] including sedimentation, solution electrical machining by transmittance through mesh cathode and anode mounted in the discharge area from the first sedimentation basin, re-sedimentation in the second sedimentation basin, thus the space between cathode and anode is filled with granular graphite material, electrically contacted with them. Thus the fine graphite provides: lighting-filtration of insoluble particles of clay sludge from highly concentrated salt solutions, activating their coagulation due to collision with them, and electrical machining of these solutions with a current density of 20 ... 50 A/dm^2 through this graphite provides low-voltage electrophoresis of insoluble sludge particles at the selected electric field strengths of 0.05; 0.1 and 0.2 V/mm and electric coagulation due to the high electric field stress in the thin edges of the graphite particles. The used low voltage between anode and cathode eliminates undesirable metal deposition on the cathode from the electrolyte. An increased total area of depositing anode and relative filling of the filtered space with graphite, electrically contacting the electrodes, provide an increase in speed of fine suspension particle coagulation in their sedimentation by 3 times. This is observed despite particles of less than 1 μm, i.e. not initially deposited by gravity, with concentration of 1 ... 20 % of the solution weight, have electrophoretic speed v_{ph} respectively not higher than $0.32\cdot10^{-2}$, $0.68\cdot10^{-2}$ and $1.37\cdot10^{2}$ cm/hour, i.e. significantly below the speed v_l > 1 m/sec of the electrolyte flow to be purified. The concentration of graphite particles or the distance at which the particles of suspensions and graphite should be located for coagulation due to thermal collisions, overcoming barriers of electrostatic repulsion and riving of wetting solution, is defined by the Derjaguin-Verwey-Overbeek-Landau equation.

[0004] However, the actual distance between the graphite particles, while the device is implementing the method, is constantly changing. Therefore, the main disadvantage of the method is current shunting passing through the filtered part of contaminated electrolyte, by a chain of mutually contacting graphite particles, or the loss of electric contact with electrodes due to delamination of the graphite particle concentration and growth of the insulating sludge layer. Due to the mutual screening of the particles, including due to the double electrical layer at the poles of induced electric dipole, which properties are acquired by these isolated particles, an electric potential difference even between two points in this area, one of which is close to cathode, and the second - to anode, becomes smaller ten times compared to the calculated value. Therefore electrophoresis becomes ineffective, and sludge electrical coagulation will only occur near anode and cathode of the device.

[0005] Another disadvantage of the device is frequent changes of the graphite material, quickly covered with sludge and cedimented. This increases the cost of electrolyte purification.

[0006] The third disadvantage of the device - the inability to use the method for purification of solutions of salts of iron and other transition metals due to their oxidation by air oxygen incoming with fresh particulate graphite and transition into insoluble sludge particles. This not only further contaminates electrolyte but removes salt from the working solution, making it unsuitable for use.

[0007] Despite two conjugate superadditive operations of solution pretreatment, they are attached to a small part of its volume and their action does not allow full removal of the sludge. Sedimentation at using a compact installation leads to its low performance. Therefore, the above method does not provide commercially attractive obtaining of low-cost *ultra-pure* electrolyte of iron and other transition metals.

[0008] The goal of the invention is to increase efficiency and quality of purification of salt aqueous solutions of iron and other transition metals, to increase compactness and commercial attraction of the electrolyte purification method and of the refiner for its application.

[0009] The goal is achieved by:

1. An electrolyte purification method, including the low-voltage electrical machining of the electrolyte flow by transmittance it through mesh cathode and anode and by sedimentation, was added with an additional preliminary electrical machining with high-density current and associated heating, superadditive relating to these electrical machining processes, followed by mechanical filtration and electrolyte measurement before, between, and after electrical machining, automatic calculation and control over electrical machining current, electrolyte temperature and flowrate, sludge removal, signal generation for bath maintenance and on the purification end, supply of a new portion of impure electrolyte, its components, and purified electrolyte discharge, thus the electrolyte is treated fully sealed and unexposed to air;

2. To apply this method, a refiner, consisting of a unit 1 for sludge pretreatment acting as a sediment basin and a low-voltage electrophoresis block, was equipped with an additional mechanical filtration unit 2 and a control unit 3; thus the pretreatment unit 1 and the mechanical filtration unit 2 are series-connected; the first input of the sludge pretreatment unit 1 is connected to the source 5 for supply of impure electrolyte and its components through the first solenoid valve 4, the output of the mechanical filtration unit 2 is connected through the second solenoid valve 6 to the drain 7 of corrected purified electrolyte and to the second input of the pretreatment unit 1, which inputs also serve as the refiner inputs, a group of the sludge removal lines from the preliminary unit 1 is connected through the first group 8 of solenoid valves to the sludge drain 9, to which the group of sludge removal lines from the mechanical filtration unit 2 is connected through the second group 10 of solenoid valves; the signal outputs of both units are connected to the inputs of the control units 3, and the control inputs of both units and the control inputs of the first solenoid valve 4, the second solenoid valve 6, groups 8 and 10 of solenoid valves - to its outputs; sections of the low-voltage electrophoresis compartment 11 in the pretreatment unit 1, transversal to the electrolyte flow direction, are completely blocked with a stack 12 of insoluble mesh electrodes, *tightly* adjacent to the inner walls of the unit 1 and making the clearance δ with its bottom 13 for sludge drain into the mechanical filtration unit 2, thus $\delta \approx$ 0.5 ... 1 % of the value h, where: *h* - height of the stack 12 of mesh electrodes containing conductive sheets, electrically dissociated by dielectric sheets 14; thus the odd conductive sheets 15 are connected to the first terminal 16 of the electrode stack 12, and the even ones 17 - to its second terminal 18; the voltage of the low-voltage power source 19, to which the terminals of the electrode stack 12 are connected, equals $U_{\mathrm{L}}^{\mathrm{ph}} < U_{\mathrm{min}}^{\mathrm{Me}}$, where $U_{\mathrm{min}}^{\mathrm{Me}}$ – the minimum voltage at which electroplating of metal from the electrolyte is conducted; thus the sludge pretreatment unit 1 is additionally equipped with an electric heater 20 (R_1), adherent to the entire outer surface of its bottom 13, with a compartment 21 of high-current electrophoresis, located between the inputs of the pretreatment unit 1 and its low-voltage electrophoresis compartment 11, containing over two pairs of soluble electrodes 22 made of metal, which salt serves as the base for the electrolyte purified of fine sludge; thus each soluble electrode 22 is placed into the bigger case 23 of acidproof dielectric fabric transmitting electrolyte; pockets 24 for sludge collection, fixed underneath the sludge pretreatment unit 1 and located at the electric heater 20 sides, the pocket 24 cavities intercommunicate the cavities of the cases 23, and their outputs serve as sludge removal lines from the pretreatment unit 1; the terminals of soluble electrodes 22, related to its first half, are coupled together and connected to the first pole of the high-current DC source 25, which polarity is changed at regular intervals, and the terminals of the remained electrodes 22 - to its second pole; the pretreatment unit 1 additionally contains three triples of optical windows 26; thus the first windows of each triple are located at the first side of the unit 1, the second ones - at the second side, the third - at the top; the first window triplet is located in a transparent compartment near the unit 1 inputs, the second one - in the similar compartment between the high-current electrophoresis chamber 21 and the low-voltage electrophoresis chamber 11, and the third one - in the similar compartment near the unit 1 outputs; *LED* and laser semiconductor modules 27 are attached outside to

the first (front) and second (upper) windows of each triple, and photodetectors 28 to measure the turbidity, color and light polarization are attached to the third windows, probe devices with digital outputs - a conductivity meter 29, an automatic digital densitometer 30, a *pH*-meter 31 and a thermometer (thermo-relay) 32 are located in the compartment near the unit 1 outputs; the mechanical filtration unit 2 consists of a pump 33, which inlet serves as the input of the mechanical filtration unit 2, and at least two (*I*, *II*, ...) parallel lines of serially connected filters 34, forming at least three filtration stages in each line, and the inputs of each line are connected to the pump 33 discharge through the third group 35 of solenoid valves of these lines, the outputs are interconnected and form the output of the mechanical filtration unit 2, the second outputs of the filters 34 form a group of sludge removal lines from the mechanical filtration unit 2; a pressure relay 36 is connected to the pump 33 as well; the control unit 3 consists of a programmable controller 37, a control console 38, an indication and annunciator panel 39; thus the controller 37 inputs are connected to the control console 38 outputs and the controller 37 data inputs serve as the inputs of the control unit 3; the first group of the controller 37 outputs is connected to the inputs of the indication and annunciator panel 39, and the second group of the outputs serves as the outputs of the control unit 3; the signaling outputs of the sludge pretreatment unit 1 are formed by outputs of the photodetectors 28, the digital conductivity meter 29, the densitometer 30, the *pH*-meter 31 and the thermometer 32; and control inputs of this unit 1 - by the control inputs of the low-voltage DC source 19, electric heater 20 (R_1, TC), high-current DC source 25, inputs of the front and upper LED and laser modules 27; the signaling outputs of the mechanical filtration unit 2 are formed by the pressure relay 36 output, and control inputs - by the control inputs of the pump 33 and of the third group of solenoid valves 35.

3. the method of electrolyte purification stipulated in clause 1, additionally contains the procedure of fine sludge particle coarsening by salt crystallization during the electrolyte.

4. to apply the method, described in clause 2, the refiner is additionally equipped with a crystallizer 40, thus the crystallizer 40 consists of a body 41 with an upwards expanding inner cavity, passing into a gasholder 42 at the top, and at the bottom - into a sludge compactor 43, which side walls are adhered by the second electric heater 44 (R_2); a silicone dispenser 45 of cold nitrogen, spaced at a h_{cr} distance from the upper edge of the body 41, thus the dispenser 45 input is connected to the source 46 for cooled nitrogen supply, the gasholder 42 output is connected through the upper hole in it to a drain 47 for warm nitrogen disposal, the crystallizer 40 output is located on the line of the upper edge of its body 41 and connected to the inputs of the sludge pretreatment unit 1, the first and second inputs of the crystallizer 40 are located below the cooled nitrogen dispenser 45 and serve as new inputs of the refiner, thus the first input is connected to the source 5 for impure electrolyte supply and its components through the first solenoid valve 4, and the second input is connected to the output of the mechanical filtration unit 2, the output of the sludge compactor 43 is located at the compactor bottom and connected to the sludge drain 23 through the third single solenoid valve 48, the control inputs of the second electric heater 44 (R_2, TC) and solenoid valve 48 are connected to the additional outputs of the control unit 3.

[0010] The essential difference of the method of removal of analogue and prototype from electrolyte is the addition of the previous electrical machining with high-density current and heating, coupled with the electrolyte electrical machining, being superadditive regarding these processes, as well as the addition of mechanical filtration, and electrolyte measurement before, between, and after electrical machining, automatic calculation and control over electrical machining current, electrolyte temperature and flowrate, automatic sludge removal, signal generation for bath maintenance and on the purification end, supply of a new portion of impure electrolyte, its components, and purified electrolyte discharge, thus the electrolyte is treated fully sealed and unexposed to air; The integrated processes combined this way, providing their superadditive interaction, are applied for the first time, therefore, indicate a novelty of the technical solution.

[0011] The essential difference of the refiner to apply the proposed electrolyte purification method is addition of a mechanical filtration unit 2 and a control unit 3; thus the sludge pretreatment unit 1 and the mechanical filtration unit 2 are series-connected; the first input of the sludge pretreatment unit 1 is connected to the source 5 for supply of impure electrolyte and its components through the first solenoid valve 4, the output of the mechanical filtration unit 2 is connected through the second solenoid valve 6 to the drain 7 of corrected purified electrolyte and to the second input of the pretreatment unit 1, which inputs also serve as the refiner inputs, a group of the sludge removal lines from the preliminary unit 1 is connected through the first group 8 of solenoid valves to the sludge drain 9, to which the group of sludge removal lines from the mechanical filtration unit 2 is connected through the second group 10 of solenoid valves; the signal outputs of both units are connected to the inputs of the control units 3, and the control inputs of both units and the control inputs of the first solenoid valve 4, the second solenoid valve 6, groups 8 and 10 of solenoid valves - to its outputs; sections of the low-voltage electrophoresis compartment 11 in the pretreatment unit 1, transversal to the electrolyte flow direction, are completely blocked with a stack 12 of insoluble mesh electrodes, *tightly* adjacent to the inner walls of the unit 1 and making the clearance δ with its bottom 13 for sludge drain into the mechanical filtration unit 2, thus $\delta \approx 0.5...1\%$ h, where: h - height of the stack 12 of mesh electrodes containing conductive sheets, electrically dissociated by dielectric sheets 14; thus the odd conductive sheets 15 are connected to the first terminal 16 of the electrode stack 12, and the even ones 17 - to its second terminal 18; the voltage of the low-voltage power source 19, to which the terminals of the electrode stack 12 are connected, equals $U_{\mathrm{L}}^{\mathrm{ph}} < U_{\mathrm{min}}^{\mathrm{Me}}$, where $U_{\mathrm{min}}^{\mathrm{Me}}$ — the minimum voltage at which electroplating of metal from the electrolyte is conducted; thus the sludge pretreatment unit 1 is additionally equipped with an electric heater 20 (R_1), adherent to the entire outer surface of its bottom 13, with a compartment 21 of high-current electrophoresis, located between the inputs of the pretreatment unit 1 and its low-voltage electrophoresis compartment 11, containing over two pairs of soluble electrodes 22 made of metal, which salt serves as the base for the electrolyte purified of fine sludge; thus each soluble electrode 22 is placed into the bigger case 23 of acidproof dielectric fabric which is permeable to the electrolyte; pockets 24 for sludge collection, fixed underneath the sludge pretreatment unit 1 and located at the electric heater 20 sides, the pocket 24 cavities intercommunicate the cavities of the cases 23, and their outputs serve as sludge removal lines from the pretreatment unit 1; the terminals of soluble electrodes 22, related to its first half, are coupled together and connected to the first pole of the high-current DC source 25, which polarity is changed at regular intervals, and the terminals of the remained electrodes 22 - to its second pole; the pretreatment unit 1 additionally contains three triples of optical windows 26; thus the first windows of each triple are located at the first side of the unit 1, the second ones - at the second side, the third - at the top; the first window triplet is located in a transparent compartment near the unit 1 inputs, the second one - in the similar compartment between the high-current electrophoresis chamber 21 and the low-voltage electrophoresis chamber 11, and the third one - in the similar compartment near the unit 1 outputs; LED and laser semiconductor modules 27 are attached outside to the first (front) and second (upper) windows of each triple, and photodetectors 28 to measure the turbidity, color and light polarization are attached to the third windows, probe devices with digital outputs - a conductivity meter 29, an automatic digital densitometer 30, a *pH*-meter 31 and a thermometer (thermo-relay) 32 are located in the compartment near the unit 1 outputs; the mechanical filtration unit 2 consists of a pump 33, which inlet serves as the input of the mechanical filtration unit 2, and at least two (*I*, *II*...) parallel lines of serially connected filters 34, forming in each line at least three filtration stages, and the inputs of each line are connected to the pump 33 discharge through the third group 35 of solenoid valves of these lines, the outputs are interconnected and form the output of the mechanical filtration unit 2, the second outputs of the filters 34 form a group of sludge removal lines from the mechanical filtration unit 2; a pressure relay 36 is connected to the pump 33 as well; the control unit 3 consists of a programmable controller 37, a control console 38, an indication and annunciator panel 39; thus the controller 37 inputs are connected to the control console 38 outputs and the controller 37 data inputs serve as the inputs of the control unit 3; the first group of the controller 37 outputs is connected to the inputs of the indication and annunciator panel 39, and the second group of the outputs serves as the outputs of the control unit 3; the signaling outputs of the sludge pretreatment unit 1 are formed by outputs of the photodetectors 28, the digital conductivity meter 29, the densitometer 30, the *pH*-meter 31 and the thermometer 32; and control inputs of the unit 1 - by the control inputs of the low-voltage DC source 19, electric heater 20 (R_1, TC), high-current DC source 25, inputs of the front and upper LED and laser modules 27; the signaling outputs of the mechanical filtration unit 2 are formed by the pressure relay 36 output, and control inputs - by the control inputs of the pump 33 and of the third group of solenoid valves 35. The integrated components and their bonds combined this way and rest components of the refiner are applied for the first time, therefore, indicate a novelty of the technical solution.

[0012] The essential difference of the second method of removal of analogue and prototype from electrolyte is an additional coarsening of fine sludge particles by salt crystallization during electrolyte cooling. The integrated operation and its interconnection with the others are applied for the first time, therefore, indicate a novelty of the technical solution.

[0013] The essential difference of the refiner to apply the second proposed method of removal of analogue and prototype from electrolyte is an additional crystallizer 40, thus the crystallizer 40 consists of a body 41 with an upwards expanding inner cavity, passing into a gasholder 42 at the top, and at the bottom - into a sludge compactor 43, which side walls are adhered by the second electric heater 44 (R_2); a silicone dispenser 45 of cold nitrogen, spaced at a h_{cr} distance from the upper edge of the body 41, thus the dispenser 45 input is connected to the source 46 for cooled nitrogen supply, the gasholder 42 output is connected through the upper hole in it to a drain 47 for warm nitrogen disposal, the crystallizer 40 output is located on the line of the upper edge of its body 41 and connected to the inputs of the sludge pretreatment unit 1, the first and second inputs of the crystallizer 40 are located below the cooled nitrogen dispenser 45 and serve as new inputs of the refiner, thus the first input is connected to the source 5 for impure electrolyte supply and its components through the first solenoid valve 4, and the second input is connected to the output of the mechanical filtration unit 2, the output of the sludge compactor 43 is located at the compactor bottom and connected to the sludge drain 23 through the third single solenoid valve 48, the control inputs of the second electric heater 44 (R_2, TC) and the solenoid valve 48 are connected to the additional outputs of the control unit 3. The integrated into the refiner components and their intercommunication with the rest components are applied for the first time, therefore, indicate a novelty of the technical solution.

[0014] Thus, the first option of the proposed method of electrolyte purification includes the electrical machining with high-density current and low-voltage electrical machining with electrolyte flow transmittance through mesh cathode and anode and associated heating, superadditive regarding these processes, mechanical filtration, and electrolyte measurement before, between, and after electrical machining, automatic calculation and control over electrical machining current, electrolyte temperature and flowrate, sludge removal, signal generation for bath maintenance and on the purification end, supply of a new portion of impure electrolyte, its components, and purified electrolyte discharge, thus the electrolyte is treated fully sealed and unexposed to air;

[0015] A block diagram of the refiner for the first method of electrolyte purifification is shown in Fig. 1 and comprises a unit 1 for sludge pretreatment acting as a sediment basin and a low-voltage electrophoresis block, a mechanical filtration unit 2 and a control unit 3; thus the pretreatment unit 1 and the mechanical filtration unit 2 are series-connected; the first input of the sludge pretreatment unit 1 is connected to the source 5 for supply of impure electrolyte and its components through the first solenoid valve 4, the output of the mechanical filtration unit 2 is connected through the second solenoid valve 6 to the drain 7 of corrected purified electrolyte and to the second input of the sludge pretreatment unit 1, which inputs also serve as the refiner inputs, a group of the sludge removal lines from the preliminary unit 1 is connected through the first group 8 of solenoid valves to the sludge drain 9, to which the group of sludge removal lines from the mechanical filtration unit 2 is connected through the second group 10 of solenoid valves; the signal outputs of both units are connected to the inputs of the control units 3, and the control inputs of both units and the control inputs of the first solenoid valve 4, the second solenoid valve 6, groups 8 and 10 of solenoid valves - to its outputs; The refiner sketch illustrating features of this design is shown in Fig. 2. According to this sketch, the refiner comprises a sludge pretreatment unit 1, acting as a sediment basin and a low-voltage electrophoresis block, a mechanical filtration unit 2 and a control unit 3; thus the pretreatment unit 1 and the mechanical filtration unit 2 are series-connected; the first input of the sludge pretreatment unit 1 is connected to the source 5 for supply of impure electrolyte and its components through the first solenoid valve 4, the output of the mechanical filtration unit 2 is connected through the second solenoid valve 6 to the drain 7 of corrected purified electrolyte and to the second input of the sludge pretreatment unit 1, which inputs also serve as the refiner inputs; sections of the low-voltage electrophoresis compartment 11 in the pretreatment unit 1, transversal to the electrolyte flow direction, are completely blocked with a stack 12 of insoluble mesh electrodes, *tightly* adjacent to the inner walls of the unit 1 and making the clearance δ with its bottom 13 for sludge drain into the mechanical filtration unit 2, thus $\delta \approx 0.5... 1\%$ h, where: h - height of the stack 12 of mesh electrodes containing conductive sheets, electrically dissociated by dielectric sheets 14; thus the odd conductive sheets 15 are connected to the first terminal 16 of the electrode stack 12, and the even ones 17 - to its second terminal 18; the voltage of the low-voltage power source 19, to which the terminals of the electrode stack 12 are connected, equals $U_{\mathrm{L}}^{\mathrm{ph}} < U_{\mathrm{min}}^{\mathrm{Me}}$, where $U_{\mathrm{min}}^{\mathrm{Me}}$ — the minimum voltage at which metal electroplating from the electrolyte is conducted; the sludge pretreatment unit 1 also comprises an electric heater 20 (R_1), adherent to the entire outer surface of its bottom 13, with a compartment 21 of high-current electrophoresis, located between the inputs of the pretreatment unit 1 and its low-voltage electrophoresis compartment 11, containing over two pairs of soluble electrodes 22 made of metal, which salt serves as the base for the electrolyte purified of fine sludge; thus each soluble electrode 22 is placed into the bigger case 23 of acidproof dielectric fabric transmitting electrolyte; pockets 24 for sludge collection, fixed underneath the sludge pretreatment unit 1 and located at the electric heater 20 sides, the pocket 24 cavities intercommunicate the cavities of the cases 23, and their outputs serve as sludge removal lines from the pretreatment unit 1; the terminals of soluble electrodes 22, related to its first half, are coupled together and connected to the first pole of the high-current DC source 25, which polarity is changed at regular intervals, and the terminals of the remained electrodes 22 - to its second pole; the pretreatment unit 1 additionally contains three triples of optical windows 26; thus the first windows of each triple are located at the first side of the unit 1, the second ones - at the second side, the third - at the top; the first window triplet is located in a transparent compartment near the unit 1 inputs, the second one - in the similar compartment between the high-current electrophoresis chamber 21 and the low-voltage electrophoresis chamber 11, and the third one - in the similar compartment near the unit 1 outputs; LED and semiconductor laser modules 27 are attached outside to the first and second windows of each triple, and photodetectors 28 to measure the turbidity, color and light polarization are attached to the third windows, probe devices with digital outputs - a conductivity meter 29, an automatic digital densitometer 30, a *pH*-meter 31 and a thermometer (thermo-relay) 32 are located in the compartment near the unit 1 outputs; the mechanical filtration unit 2 consists of a pump 33, which inlet serves as the input of the mechanical filtration unit 2, and at least two *(I*, *II*, ...) parallel lines of serially connected filters 34, forming in each line at least three filtration stages, thus the inputs of each line are connected to the pump 33 discharge through the third group 35 of solenoid valves of these lines, the outputs are interconnected and form the output of the mechanical filtration unit 2, the second outputs of the filters 34 are connected to the sludge drain 9 through the third group 35 of solenoid valves forming a group of sludge removal lines from the mechanical filtration unit 2; a pressure relay 36 is connected to the pump 33 as well; the control unit 3 comprises a programmable controller 37, a control console 38, an indication and annunciator panel 39; thus the controller 37 inputs are connected to the control console 38 outputs, the controller 37 data inputs serve as the inputs of the control unit 3, to which outputs of the photodetectors 28, the digital conductivity meter 29, the densitometer 30, the *pH*-meter 31 and the thermometer 32 are connected, serving the signaling outputs of the sludge pretreatment unit 1, and also the pressure relay 36 output serving the signal outputs of the mechanical filtration unit 2; the first group of the controller 37 outputs is connected to the inputs of the indication and annunciator panel 39 and the second group of outputs serve as inputs of the control units 3, to which the control inputs of the first solenoid valve 4, the second solenoid valve 6, groups 8 and 10 of solenoid valves are connected, the control inputs of the low-voltage DC source 19, the electric heater 20 (R_1, TC), the high-current DC source 25, inputs of the front and upper LED and laser modules 27 serving as inputs of the sludge pretreatment unit 1, and the inputs of the pump 33 and of the third group of solenoid valves 35 serving as the control inputs of the mechanical filtration unit 2.

[0016] The second method of electrolyte purification includes coarsening of fine sludge particles by salt crystallization during electrolyte cooling, high-density current electrical machining, low-voltage electrical machining with electrolyte transmittance through mesh cathode and anode and associated heating, superadditive relating to these electrical machining processes, mechanical filtration and electrolyte measurement before, between, and after electrical machining, automatic calculation and control over electrical machining current, electrolyte temperature and flowrate, sludge removal, signal generation for bath maintenance and on the purification end, supply of a new portion of impure electrolyte, its components, and purified electrolyte discharge, thus the electrolyte is treated fully sealed and unexposed to air.

[0017] The refiner sketch for application of the second method of electrolyte purification is shown in Fig. 3. According to this sketch, the refiner comprises a sludge pretreatment unit 1, acting as a sediment basin and a low-voltage electrophoresis block, the mechanical filtration unit 2 and the control unit 3, and the crystallizer 40, thus the sludge pretreatment unit 1 and the mechanical filtration unit 2 are series-connected and connected to the crystallizer 40 output, two inputs of which are new outputs of the refiner, thus the first input of the crystallizer 40 is connected to the source 5 for impure electrolyte supply and its components through the first solenoid valve 4, and the second input is connected to the output of the mechanical filtration unit 2, which is connected through the second solenoid valve 6 to the drain 7 of corrected purified electrolyte, a group of the sludge removal lines from the preliminary unit 1 is connected through the first group 8 of solenoid valves to the sludge drain 9, to which the group of sludge removal lines from the mechanical filtration unit 2 is connected through the second group 10 of solenoid valves; the signal outputs of the preliminary unit 1 and the mechanical filtration unit 2 are connected to the inputs of the control units 3; the crystallizer 40 consists of a body 41 with the upwards expanding inner cavity, continued by the gasholder 42 narrowing at the top, and at the bottom - by the sludge compactor 43, which side walls are adhered by the second electric heater 44 (R_2); the silicone dispenser 45 of cold nitrogen, spaced at the h_{cr} distance from the upper edge of the body 41, thus the dispenser 45 input is connected to the source 46 for cooled nitrogen supply, the gasholder 42 output is connected through the upper hole in it to a drain 47 for warm nitrogen disposal, the crystallizer 40 output is located on the line of the upper edge of its body 41 and its first and second inputs are located below the cooled nitrogen dispenser 45, the output of the sludge compactor 43 is located at the compactor bottom and connected to the sludge drain 9 through the third single solenoid valve 48, the control inputs of the second electric heater 44 (R_2, *TC*) and solenoid valve 48 are connected to the additional outputs of the control unit 3, and the control inputs of the sludge pretreatment unit 1, the mechanical filtration unit 2, the control inputs of the first solenoid valve 4, the second solenoid valve 6, groups 8 and 10 of solenoid valves and the third solenoid valve 48 - to its outputs.

[0018] The refiner function algorithm for the first electrolyte purification method, which block diagram is shown in Fig. 4, is carried out as follows.

[0019] The fine sludge is considered as the most difficult to be removed from electrolytes, based on transition metal salts, quickly oxidized while exposed to air, contained in these insoluble colloidal particles. A saturated aqueous solution of ferric chloride $FeCl_2$ is a typical representative of these imbalance electrolytes, with ultra purity to be achieved is believed to be the most commercially attractive and of the greatest practical significance [26]. Purification and correction of electrolytes, based on salts of other transition metals, with the proposed methods and refiners for their application, differ only by trigger threshold setting of inputs of the control unit 3 and using electrodes 22 in the high-current electrophoresis compartment 21 of the sludge pretreatment unit 1 made of metal, corresponding to these electrolytes. Therefore, the following description is related only to an electrolyte with the most evident characteristics of sludging-up and the same methods of sludging-up inherent to electrolytes of other transition metal - saturated aqueous solution of $FeCl_2$ and to the electrolyte, respectively, - the electrodes 22 of unalloyed iron (Armco). When $FeCl_2$ salt exposed to air, it is oxidized forming insoluble particles of oxychlorides $FeOCl$ [27].

[0020] The method is provided by simultaneous physical-chemical processes, i.e. electrolyte heating, high-current electrophoresis, low-voltage electrophoresis, and mechanical filtration. At the electrolyte temperature of above 75 °C, the frequency and energy of collision of fine sludge particles are sufficient for adhesion of particles of greater than 200 nm. This is evident even visually by quick turbidity of impure electrolyte [28]. Simultaneous electrophoresis processes lead to increased colloidal sludge concentration near the electrodes, and the particle coarsening by the electrolyte heating is accelerated more than 10 ... 100 times. Thus the coarsening involves a big part of all insoluble particles, i.e. particles of greater than 1 nm. The associated high-current electrophoresis causes polarization and ionization of sludge particles, hence their electrical coagulation, passing into flocullation. Therefore, increasing in the sludge concentration near the electrodes and particle coarsening are accelerated more than 10 ... 100 times. Large particles, while colliding, capture finer ones, and the resulting electrolyte clarification near the electrodes is observed even visually. Obviously, thus the desired superadditive result of sludge coarsening is achieved, representing not the cumulative result of the used physicochemical processes, but due to the process conjugation with their mutual reinforced actions, it is k_{sa} = 100 ... 10'000 times more effective than this sum by the rate, by reduced concentration of the non-coarsen sludge, which can not be removed from the electrolyte by the mechanical filtration unit 2. The low-voltage electrophoresis functions in the low-voltage electrophoresis compartment 11 is similar. Even the usual combination of several functions of one unit capacity allows to reduce its dimensions and to increase its productivity and efficiency, including the quality of the device functions. Superadditive improves the device parameters by k_{sa} times.

[0021] The combination of two types of electrophoresis is required to improve the refiner performance, to reduce energy costs for electrolyte treatment by automatic optimization of the schedule of the use of capabilities of these types of electrophoresis. The high-current electrophoresis is especially effective at an initial stage of sludge removal and correction of the electrolyte composition, since it allows not only to coarsen the sludge quickly, but also to restore the ion concentration of the working metal in the electrolyte, decreased as a result of its use in chemical and metal electroplating, as well as of oxidation when exposed to air. Traditional electroplating technology is limited only by this kind of electrolyte treatment, its duration in hours is approximately determined from the empirical relation T ≈ (12 ... 14) V/I, where: V - the treated electrolyte volume in liters, I - electric current flowing through the electrolyte volume V [29], [3], [30], [13]. However, for each volume V_1 = 100 liter at current $I_{\mathrm{H}}^{\mathrm{ph}} = 20$ A, the effectiveness of this type of electrical-machining decreases 6 times in 3 hours, and electric energy will be mainly spent for the electrolyte ohmic heating, even though the colloidal sludge concentration in electrolyte is decreased only by 50 %. Thus, depending on the electrodes 22 surface, the density $J_{\mathrm{H}}^{\mathrm{ph}}$ of current near cathode and anode surfaces can be selected from a range of 20 ... 350 A/dm^2. Therefore, growing on the group 22 electrode cathode surface of multiple branching growths - brittle dendrites, mainly formed by deposited metal and sludge captured by electrophoresis - is also typical for the high current DC electrical machining. Since the length of the dendrit branches exceeds 3 cm in 3 ... 4 hours, further the dendrites grow through the case 23 and reach anodes, causing a short-circuit of the power source 25.

[0022] Meanwhile anodes from the electrode 22 group almost completely dissolve and become unsuitable for further use. To prevent these undesirable events, an automatic change of the output polarity in the high power source 25 is provided at regular intervals ΔT ≈ 5 ... 30 minutes. Obviously, the electrodes 22 metal in this case is consumed insignificantly - only to restore the ion concentration in the electrolyte, which typically decreases by less 2 %. Since the necessity to restore the metal ion concentration in the electrolyte disappears in 3 ... 4 hours, then only low-voltage electrophoresis is advisable to be used in the future to save energy. The low voltage is required between the fabric electrodes 15 and 17 of the electrode stack 12 because the metal deposition is unacceptable since it could cause not only their short circuit, but also the loss of electrode porosity and electrolyte clog in the compartment 11 volume. Since transition metals in aqueous solutions are mainly electronegative, i.e. are located to the left of hydrogen in electrochemical series, the voltage on the electrodes 15 and 17 can be reduced to values when the metal isolation on cathode becomes impossible. However thanks to the close alternating fabric electrodes 15 and 17, separated only by a thin dielectric sheet and covering the whole cross section of electrolyte flow, the sludge particles way decreases in almost the entire volume of the low-voltage electrophoresis compartment 11, accelerating their coarsening and sedimentation, even at drops of the low-voltage supply 19 voltage $U_{\mathrm{L}}^{\mathrm{ph}}$ to values of 0,025 V. The low-voltage electrophoresis chamber 11 operation remains efficient, as coagulation occurs in most colloids at the isoelectric potential of 0,025 ... 0.03 V (see [23], pg. 220 [33] [34]). This voltage can be increased to Д o $U_{\mathrm{L}}^{\mathrm{ph}} \leq 0{,}441$ V at the treatment of $FeCl_2$ salt aqueous solution.

[0023] Thanks to coagulation of the most colloidal sludge, and the sludge not forming colloidal particles, the obtained coarsen sludge can be removed from the electrolyte using available highly-efficient mechanical filters periodically cleaned of accumulated sludge, having an inexpensive structure based on cartridges with pores of 1 ... 5 μm. To ensure high performance and better removal of the remaining fine particles mainly of 1 ... 10 nm, the electrolyte returns to repeat the treatment and purification cycles, after the first treatment cycle, through the connected output of the mechanical filtration unit 2 and the input of the sludge pretreatment unit 1. This allows to remove the sludge, more stable due to its decreased concentration and concentration of particles tended to coagulate. Therefore, the used superadditive processes of sludge coagulation are non-selective, affecting all kinds of fine sludge, irrespective of dispersion, structure and chemical composition.

[0024] Ultra-pure electrolyte with high performance and cost minimization is available thanks to the automatic control of the electrolyte parameters and the timely automatic switching of sludge treatment and filtration modes, correction of electrolyte composition. This function is performed by the refiner control unit 3 operating as follows.

[0025] Once the refiner is turned on, the 3 control unit is initialized, and the signals are continuously sampled from outputs of the photodetector 28 to measure the turbidity, color, and light polarization, and the thermal relay 32 during alternating pulse switching on of the front LED and laser modules 27, and alternating sequential pulsed switchings on of the upper LED and laser modules 27. If the electrolyte amount in the refiner is insufficient for its normal operation, the signal at the photodetectors 28 outputs is maximal for a horizontal beam from the laser modules 27 with vertical light polarization, and is absent at the photodetector 28 outputs for a scattered beam from the upper laser modules 27 with polarization by normal to the angle of the beam, no color signal and perpendicular components of the light polarization appear at the photodetector 28 outputs. In this case, the controller 37 sends to the indication and annunciator panel 39 a command to display signs to fill electrolyte, sets a command to open the first solenoid valve 4 and continues the mode of initial sample of the photodetector 28. Thus, the control unit 3 monitors the electrolyte level in the refiner. In this mode, its commands for the rest of the controlled refiner components are not active.

[0026] If the sludge pretreatment unit 1 contains the required amount of impure electrolyte, the same low signal is set at the photodetector 28 outputs. Then signals to turn on these power sources are set at the control unit 3 outputs, connected to the control inputs of the low-voltage source 19 and the high-current source 25, the signal to open the valve 4 is switched off. At the same time the control unit 3 constantly checks the thermal relay 32 output state, and if the contacts are not closed or an electrolyte temperature signal is below a predetermined value of 75 ° ... 90 °C, the control unit 3 sends a command to switch the first electric heater 20 (R_1, TC) on; later when the thermal relay 32 contacts are closed or a temperature signal is higher than predetermined, this command is always switched off and on, when the thermal relays 32 outputs return into the initial state. If the electrolyte temperature is not decreased over time T_{cool} > 1 min at the switched off electric heater 20, then the high-current source 25 can be switched off to reduce the electrolyte temperature, however after achieved electrolyte temperature decrease below predetermined of 75 ...°90°°C, a program jump of the control unit 3 occurs not to enable the electric heater 20, but to switch off the high-current source 25. Only a heating delay more than T_{heat} > 1 min to temperature higher the threshold will cause switching on of the electric heater 20. This allows to maintain a constant electrolyte temperature, rationally using heat from high current electrophoresis. Once the first electrolyte temperature setpoint is reached, the control unit 3 sends a command to switch the pump 33 on and the valve of the third group of valves 35, relating to the first input line I of the filters 34.

[0027] Output signals of photodetectors 28 are changed during the sludge treatment in the sludge pretreatment unit 1 and its removal in the mechanical filtration unit 2, allowing to measure the sludge concentration at different stages of removal of sludge:

- at the beginning - rough, turbidity by turbidimetry (transmitted light absorption) and nephelometry (light scattering by a turbid medium) (see [35], [2], pg. 346 [36] [37], [38]);

- at the phase of chemically pure electrolyte - by measuring the intensity of the scattered polarized light [39] (brightness measurement by Tyndall cone, i.e. nephelometry);

- then - by measuring the intensity of the perpendicular component of the linearly-polarized light polarization [40] and [41] (measurement of full transparency, i.e. turbidimetry);

- at the phase of determination of ultra-pure electrolyte parameters - by measuring changes in the spectral composition of the transmitted and scattered light [42], [43]. At this final stage of ultra-pure electrolyte $FeCl_2$, getting, its color in the Tyndall effect fully loses a brown component, which causes a mixed green color in slime electrolyte, even transparent. Just like in many solutions of colloidal colouring agents, the color is changed into spectrally complementary color at strong dilution, the colour of aqueous solution of $FeCl_2$ becomes the same as in crystals $FeCl_2 \cdot 4H_2O$, i.e. almost disappearing, with a slight blue shade [1], [44]. In almost full absebce of hydrated colouring sludge particles, i.e. at sludge concentration of 10^{-6} ... 10^{-8} % [45], $FeCl_2$ aqueous solution becomes colorless. Similarly, the color disappears at purification of the copper salts solution, i.e. solutions of monovalent copper ions. The color of salt solutions of other metals, associated with ions of the selected valency for these metals, does not disappear by removing fine sludge; however it alters the spectral composition of the reflected scattered (Tyndall) and transmitted light, eliminates the perpendicular component in the polarization of the scattered light associated with the sludge. Therefore, light filters of the photodetectors 28, corresponding to the spectral clear composition of the metal ions, are used at purification of salt solution of other metals.

[0028] These measurement methods with the LED and laser modules 27 and the photodetectors 28 are automatically changed in the control unit 3 when the photodetectors 28 signal levels are changed; it also allows to determine automatically the precipitated sludge amount and accordingly - the time to switch off the valve 35 of the first line I input of the filters 34, the time to switch on the valve 35 of the second line *II* input of the filters 34, the time to switch off the valve 35 of the second line *II* input of the filters 34 and the time to switch on the valve 35 of the third line *III* input of the filters 34, etc., the time to switch on the groups 8 and 10 valves for cleaning the pockets 24 and the lines (*I*, *II*, ..., or others) of the filters 34 of filled thick slush of insoluble particles and electrolyte. The correct signal for the sludge discharge is a signal of the pressure relay 36 setting during the filters 34 filling.

[0029] The correction of electrolyte concentration and composition, changed at its sludging-up, is followed by the procedure of the last electrolyte purification phase in the control unit 3 program. The control unit 3 polls signals at the outputs of the conductivity meter 29, the densitometer 30, the *pH*-meter 31 and the thermometer (thermorelay) 32, and, calculating the procedure parameters, sets information on the number of missing electrolyte components, i.e. acid or alkali, at the indication and annunciator panel 39, sets a signal to open the solenoid valve 4.

[0030] The theory of computing of concentration of salts and other electrolyte components, its electrochemical reactivity and density, using values of the electroconductivity, density, and hydrogen index *pH*, measured at predetermined temperature, is described in [46], [47], [48].

[0031] The true sludge-free solution of metal salt is provided by the refiner operation.

[0032] Thus the invention goal is achieved by the proposed method of electrolyte purification and the refiner to apply it - to increase efficiency and quality of purification of salt aqueous solutions of iron and other transition metals, to increase compactness and commercial attraction of the electrolyte purification method and the refiner to apply it.

[0033] The refiner function algorithm for the second electrolyte purification method, which block diagram is shown in Fig. 5, is supplemented with a procedure of fine sludge particle coarsening by salt crystallization during supersaturated electrolyte cooling, and this procedure starts the first phase of repetitive cycles of sludge treatment and electrolyte filtration. Thanks to this operation, electrolyte is quickly purified of fine particles, not tended to coagulation due to the absence of ionization, electrical polarity or the possibility of its induction, unpaired magnetic moments, hidden by an energy penetrable double electric or shielding layer in the coagulated particles. The particles formed by non-hydratable oxides, fluorides, oxychlorides, allotropic graphite modifications, hydrocarbons, and other organic compounds are characterized by the highest concentration among fine particles not tended to coagulation. These particles as well as colloidal ones are considered to be the salt crystallization centers during cooling of supersaturated aqueous solution of $FeCl_2$ and of salts of other transition metals. The resulting turbidity of fine crystals is precipitated, takes the sludge from the solution, and increases the sludge concentration to values at which coagulation is possible for the particles unable to coagulate because of their insufficient concentration in solution. Collision of sedimented crystals and remaining fine sludge particles is similar to the sorbent action, i.e. leads to the capture of fine particles and additional electrolyte purification, i.e. clarification. Thus superadditive interaction of purification by crystallization and clarification is demonstrated, which allows to remove the finest sludge, irrespective of its origin, structure and chemical composition. The sludge particles are coarsened also thanks to the partial cementation with electrolyte components, in an amount not more 0.01°% of the amount in the electrolyte. The remaining crystallized salt turns into solution again by the sludge heating. The crystallization procedure on removal of the sludge particles not tended to coagulation can reduce the number of repeated purification cycles and additionally increase in the refiner performance by 3 ...°4°times.

[0034] In accordance with the algorithm in Fig. 5, the refiner performing the second supplemented electrolyte purification method, presented in Fig. 3, operates as follows.

[0035] Once the refiner is turned on, the control unit 3 is initialized, and similarly the signals from outputs of the turbidity, color and light polarization photodetector 28 and the thermal relay 32 are continuously polled during alternating pulse switching on of the front LED and laser modules 27 and alternating sequential pulsed switchings on of the upper LED and laser module 27. If the electrolyte amount in the refiner, i.e. in the series-connected crystallizer 40, the sludge pretreatment unit 1 and the selected first line I of the filters of the mechanical filtration unit 2, is not sufficient for its normal operation, then similarly by the photodetectors 28 signals, the controller 37 of the unit 3 displays a command on the panel 39 to fill electrolyte, sets a command to open the first solenoid valve 4 of the crystallizer 40, and continues the mode of the initial poll of the photodetectors 28, constantly monitoring the electrolyte level in the refiner. Thus the commands is for the rest controlled components of the refiner remain inactive. In other respects, the algorithm of the control unit 3, based on a comparison of the measured electrolyte parameters with its tabulated values, is similar to the unit operation in the first option of the refiner except that a command to switch on the solenoid valve 43, of similar designation, of the crystallizer 40, is set with the short-term signal to switch on the solenoid valves of the group 8 of the sludge drain from the pockets 24 of the sludge pretreatment unit 1. The electric heater 44 (R_2) operates synchroniously with the electric heater 20 of the sludge pretreatment unit 1. Obviously, the refiner self-stabilization, achieved thanks to the control unit 3, excludes the need for time-consuming manual calculations and the refiner mode control, arising from differences of contamination in different electrolyte batches, to provide determined electrolyte ultra-purity with minimal energy consumption.

[0036] The following steps are carried out during the sludge removal in the crystallizer 40. The electrolyte flow in the case 43 of the crystallizer 40 is directed upwardly from its inlet to its outlet. Small bubbles of cold reactionless gas, i.e. nitrogen, rising in the electrolyte column from the distributor 45 in the form of a net of silicone pipes with small holes located above the crystallizer 40 inlet, cool electrolyte by 20... 40°C, turning it into supersaturated solution state. Fine sludge particles, to be removed from electrolyte, are considered to be crystallization nucleation arising in this case. The time T_{crist} is required to be spent to coarsen crystals till their weight sufficient for sediment, overcoming the Brownian motion in the electrolyte, flush up by the electrolyte flow and collision with nitrogen bubbles. During this period of time, these crystals, getting heavier, should only rise to a height $h_{cr} < h_{cr\ max}$, where: $h_{cr\ max}$ - the vertical distance from the distributor 45 to the crystallizer 40 outlet. To reduce the height $h_{cr\ max}$, and hence the size of the crystallizer 40 by decreasing the electrolyte flow rate, the cross-sectional area S_{cr} of the inner cavity of its case is increasing till the level h_{cr}. Due to the sludge compactor 43 heating by the electric heater 44 (R_2) to 95 ... 100 °C, less than a boiling temperature of 106 °C, the electrolyte becomes unsaturated in and on the compactor, up to the of the distributor 45 level, so the salt crystals, collected in the sludge compactor 43, are dissolved in the electrolyte, turning it into saturated state, and the heated sludge compacted and coagulates by force. The nitrogen, heated in the electrolyte, collected in the gasholder 42 of the refiner 40, is coming through the disposal drain 47 and after cooling to the cold nitrogen source 46. Re-use of nitrogen, fully purified of remains of oxygen due to the interaction with the electrolyte, allows to avoid commercial nitrogen, which contamination with oxygen achieves 2 % and, consequently, constant consumption of working salt for formation of fine sludge particles from non-hydratable, i.e. water-insoluble oxychloride *FeOCl.*

[0037] The theory of computing of the rate of the formed crystal crystallization and sedimentation in the countercurrent crystallization column is described in [45], [49], [50], [51], [52], [53], [54].

[0038] An initial solution to be purified, obtained by dissolving of available salt $FeCl_2$ ($FeCl_2 \cdot 4H_2O$ crystalline) or iron etching with hydrochloric acid, has pale green color. The refiner operation results in the deep-purified solution $FeCl_2$, acidified to $pH = 2$ with hydrochloric acid, which is transparent and colorless, acquiring a slight blue tint at pouring, despite its high concentration to 430... 650 *g/l,* i.e. results in the true solution of the high purity salt.

[0039] Thus, the proposed methods of electrolyte purification and the refiners provide achievement of the invention goal - to improve efficiency and quality of purification of salt aqueous solutions of iron and other transition metals, to increase compactness and commercial attraction of the electrolyte purification process and the refiner to apply it. The great decrease in the electrolyte cost is achieved not by reducing the electrolyte purification quality but by using automatic refiners applying the proposed treatments and purification.

[0040] The claimed methods of electrolyte purification and the refiners to apply it are believed to be universal and can be used in the production of high-pure commercial salts of transition and other metals, as well as in removal of fine sludge from mineral and fresh water without losing their healthful mineral composition, in the preparation of high-pure solutions for medical application. Due to deep purification, the refiners can be used to produce commercially attractive coatings and foils of very extensive range of pure metals not only by electroplating, but chemical deposition. An ability to customize the refiners for purification and correction of salt solutions of different metals allows their use in the production of high-quality polymetallic, coatings of alternating layers of different metals, highly corrosion resistance coatings. The refiners can also be used in the production of high-pure chemicals.

[0041] For the production of fabric electrodes 15 and 17 of the low-voltage electrophoresis chamber 11 in the sludge pretreatment unit 1, carbon fabric is used, and glass fabric - as the separation porous insulation 14. The distributor 45 of the crystallizer 40 is made in the form of a net of silicone pipes with small holes. The pretreatment unit 1 case and the crystallizer 40 are manufactured of pure titanium coated with titanium oxide or other chemically resistant material.

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List of International Patent Classification discussed in the Patent Claim:

[0043]

B01 D 8/00, 9/02, 15/40, 17/00, 17/025, 17/035, 21/00, 21/01, 21/02, 21/08, 21/28; 21/30, 21/32, 24/00, 24/10, 25/00, 27/00, 29/00, 29/50, 29/52, 29/94, 29/96, 33/44, 35 / 12, 37/00, 46/04, 51/02, 57/02, 61/04, 61/16, 61/18, 61/20;
B03B 1/02, 13/00, 13/02, 13/04, 3/00;
C02F 1/02, 1/46, 1/52, 1/463, 1/469;
C25B 15/08;
C 25D 13/24, 21/00, 21/02, 21/06, 21/12, 21/14, 21/16, 21/18;
C25F 7/00;
C30B 29/00, 35/00.

Claims

1. An electrolyte purification method, including the low-voltage electrical machining of the electrolyte flow by passing it through mesh cathode and anode and by sedimentation, **characterizing by** the additional electrical machining with high-density current before the low-voltage electrical machining with sedimentation, and associated heating, superadditive relating to these electrical machining processes, followed by mechanical filtration and electrolyte measurement before, between, and after electrical machining, automatic calculation and control over electrical machining current, electrolyte temperature and flowrate, sludge removal, signal generation for bath maintenance and on the purification end, supply of a new portion of impure electrolyte, its components, and purified electrolyte discharge, thus the electrolyte is treated fully sealed and unexposed to air.

2. A refiner for implementation of the method mentioned in clause 1, consisting of the unit for pretreatment by low-voltage electrophoresis and sedimentation, **characterizing by** an additional mechanical filtration unit and a control unit; thus the pretreatment unit and the mechanical filtration unit are series-connected; the first input of the pretreatment unit is connected to the source of impure electrolyte and its components through the first solenoid valve, the output of the mechanical filtration unit is connected through the second solenoid valve to the prepared electrolyte discharge and to the second input of the pretreatment unit, which inputs also serve as the refiner inputs, a group of the sludge removal lines from the preliminary unit is connected through the first group of solenoid valves to the sludge drain, to which the group of sludge removal lines from the mechanical filtration unit is connected through the second group of solenoid valves; the signal outputs of both units are connected to the inputs of the control units, and the control inputs of both units and the control inputs of the first solenoid valve, the second solenoid valve, both groups of solenoid valves - to its outputs; sections of the low-voltage electrophoresis compartment in the pretreatment unit, transversal to the electrolyte flow direction, are completely blocked with a stack of insoluble mesh electrodes tightly adjacent to the inner walls of this unit and making the clearance δ with its bottom for sludge drain into the mechanical filtration unit, and $\delta \approx 0.5 \ldots 1$ % h, where: h is a height of the stack of mesh electrodes containing conductive sheets, electrically dissociated by dielectric sheets; thus the odd conductive sheets are connected to the first terminal of the electrode stack, and the even ones - to its second terminal; the voltage of the low-voltage power source, to which the terminals of the electrode stack are connected, equals $U_{L}^{ph} < U_{min}^{Me}$, where Д the minimum voltage at which electroplating of metal from the electrolyte is conducted;

the pretreatment unit is additionally equipped with an electric heater R_1, adherent to the entire outer surface of its bottom, with a compartment of high-current electrophoresis, located between the inputs of the pretreatment unit and its low-voltage electrophoresis compartment, containing over two pairs of soluble electrodes made of metal, which salt serves as the base for the electrolyte; each soluble electrode is covered with acidproof dielectric fabric; pockets for sludge, fixed underneath the pretreatment unit and located without contact at the electric heater sides, thus the pocket cavities continue the cavities of the covers, and their outputs serve as sludge removal lines from the pretreatment unit; the terminals of the first half of soluble electrodes are coupled together and connected to the first pole of the high-current DC source, which polarity is changed at regular intervals, and the terminals of the second half - to its second pole;

the pretreatment unit additionally contains three triples of optical windows; the first windows of each triple are located at the first side of the unit, the second ones - at the second side, the third - at the top; the first window triplet is located in a transparent compartment near the unit inputs, the second one - in the similar compartment between the chambers for the high-current and low-voltage electrophoresis, and the third one - in the similar compartment near the unit outputs; LED and laser semiconductor modules are attached outside to the first (front) and second (upper) windows of each triple, and photodetectors are attached to the third windows, digital conductivity meter, densitometer, *pH*-meter and thermometer (thermo-relay) are located in the compartment near the unit outputs;

the mechanical filtration unit consists of a pump, which inlet serves as the unit input, and at least two (*I*, *II*, ...) parallel lines of serially connected filters, forming in each line at least three filtration stages, and the inputs of each line are connected to the pump discharge through the third group of solenoid valves of these lines, the outputs are interconnected and form the output of the mechanical filtration unit, the second outputs of the filters form a group of sludge removal lines from the mechanical filtration unit; a pressure relay is connected to the pump as well;

the control unit consists of a programmable controller, control console, indication and annunciator panel; thus the controller inputs are connected to the control console outputs and the controller data inputs serve as the inputs of the control unit; the first group of the controller outputs is connected to the inputs of the indication and annunciator panel, and the second group of the outputs serves as the outputs of the control unit;

the signaling outputs of the sludge pretreatment unit are formed by outputs of the photodetectors, digital conductivity meter, densitometer, *pH*-meter and thermometer; and control inputs of this unit - by the control inputs of the low-voltage DC source, electric heater R_1, high-current DC source, inputs of the front and upper LED and laser modules,

the signaling outputs of the mechanical filtration unit are formed by the pressure relay output, and control inputs - by the control inputs of the pump and of the third group of solenoid valves.

3. The method of electrolyte purification according to clause 1, **characterizing by** additional preliminary separation of the fine sludge by salt crystallization in its particles during the electrolyte transfer into a supersaturated state.

4. The refiner according to clause 1 **characterizing by** an additional crystallizer to perform the method stipulated in clause 3, thus the crystallizer consists of the upwards expanding body, passing into the gasholder at the top, and at the bottom - into the sludge compactor, which side walls are adhered by the second electric heater R_2; the silicone nitrogen dispenser, spaced at a h_{cr} distance from the upper edge of the body, thus the dispenser input is connected to the cooled nitrogen supply, the gasholder output is connected through the upper hole in it to a warm nitrogen drain,

the crystallizer output is located on the line of the upper edge of its body and connected to the inputs of the pretreatment unit, the first and second inputs of the crystallizer are located below the nitrogen dispenser and serve as inputs of the new refiner, thus the first input is connected to the supply of impure electrolyte and its components through the first solenoid valve, and the second input is connected to the output of the mechanical filtration unit,

the output of the sludge compactor is located at the compactor bottom and connected to the sludge drain through the third solenoid valve,

the control inputs of the second electric heater R_2, and of the third solenoid valve are connected to the additional outputs of the control unit.

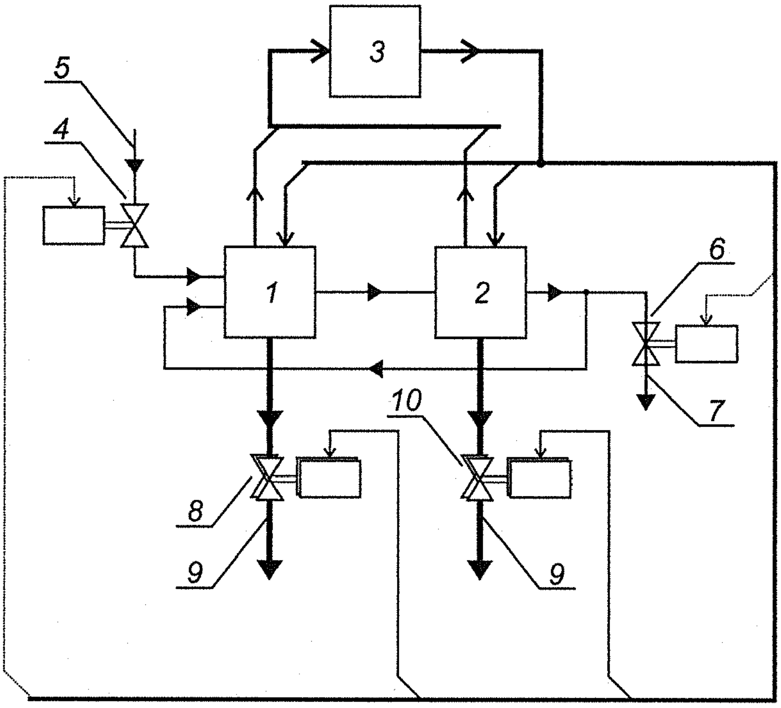

Fig. 1.

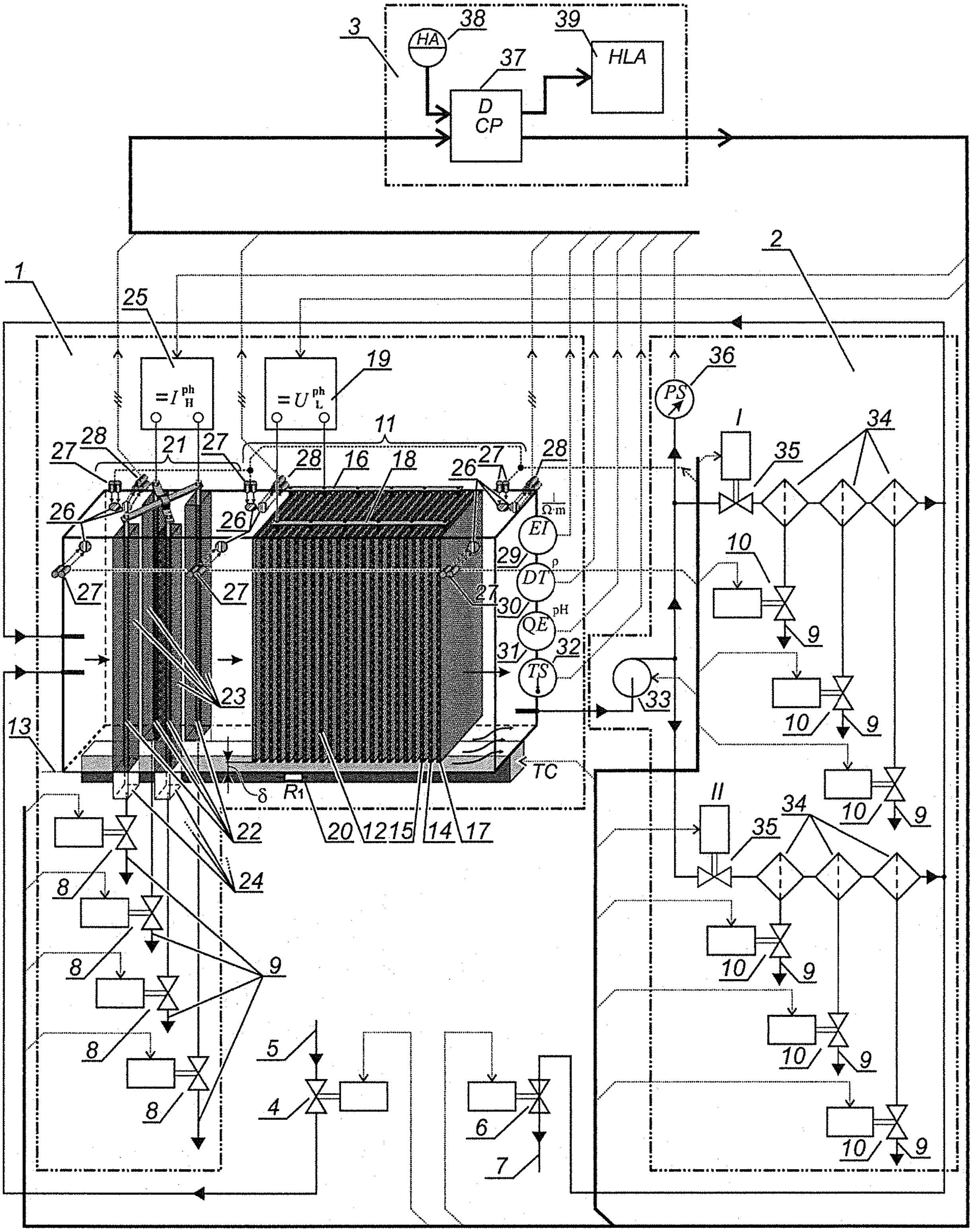
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Fig. 2.

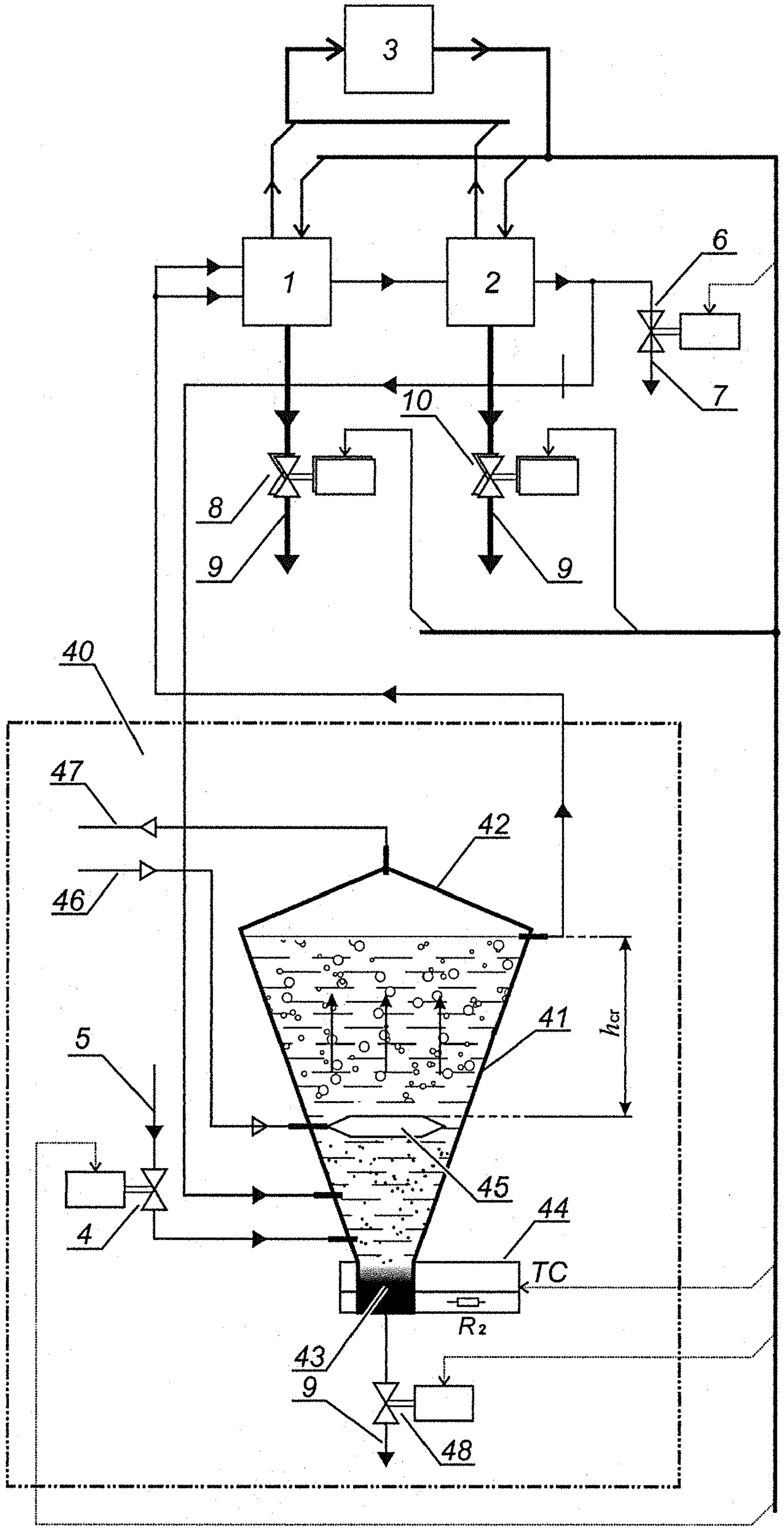

Fig. 3.

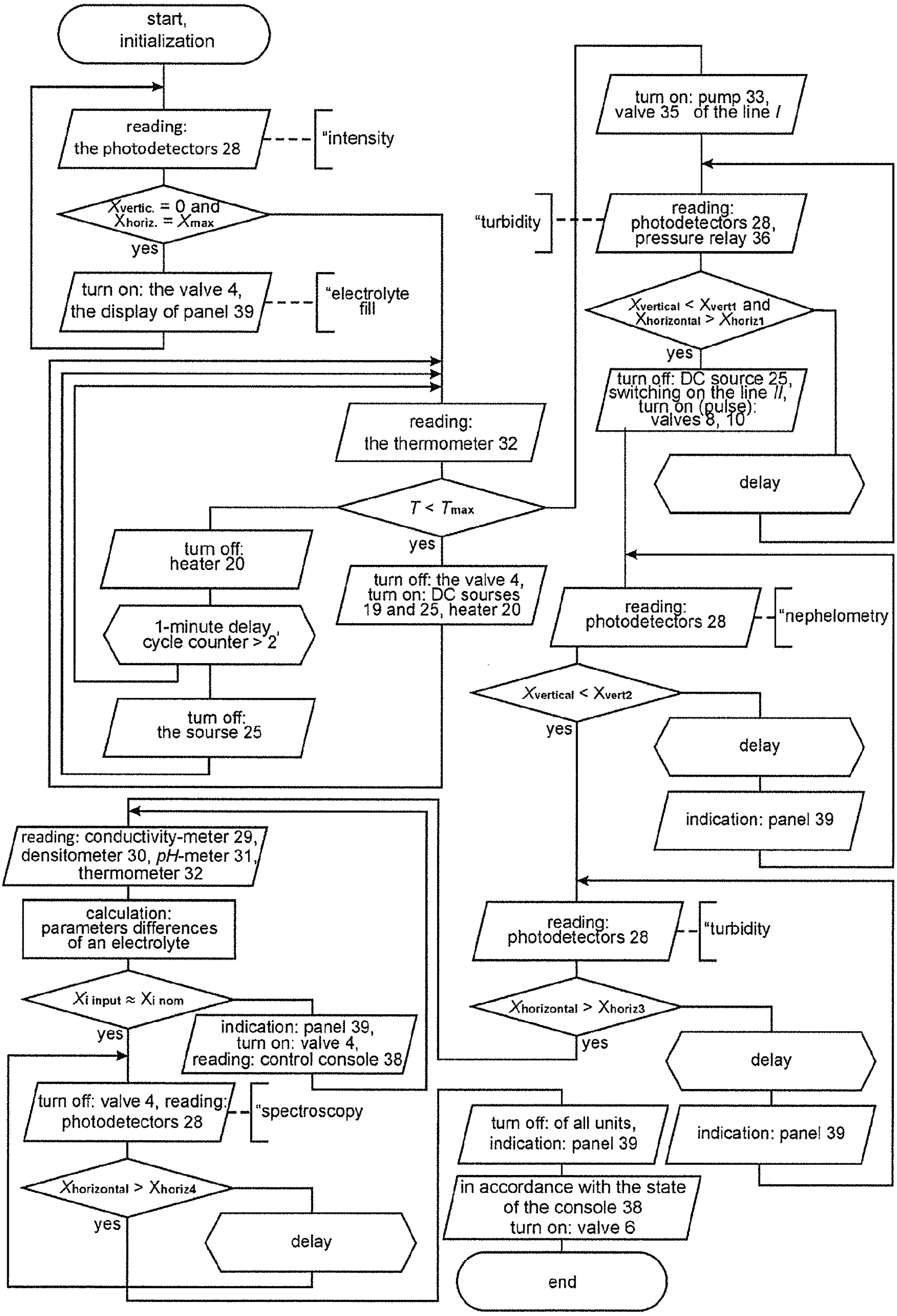
start, initialization
reading: the photodetectors 28
"intensity
Xvertic. = 0 and Xhoriz. = Xmax
yes
turn on: the valve 4, the display of panel 39
"electrolyte fill
reading: the thermometer 32
T < Tmax
yes
turn off: heater 20
1-minute delay, cycle counter > 2'
turn off: the sourse 25
turn off: the valve 4, turn on: DC sourses 19 and 25, heater 20
turn on: pump 33, valve 35 of the line I
"turbidity
reading: photodetectors 28, pressure relay 36
Xvertical < Xvert1 and Xhorizontal > Xhoriz1
yes
turn off: DC source 25, switching on the line II, turn on (pulse): valves 8, 10
delay
reading: photodetectors 28
"nephelometry
Xvertical < Xvert2
yes
delay
indication: panel 39
reading: photodetectors 28
"turbidity
Xhorizontal > Xhoriz3
yes
delay
indication: panel 39
reading: conductivity-meter 29, densitometer 30, pH-meter 31, thermometer 32
calculation: parameters differences of an electrolyte
Xi input ≈ Xi nom
yes
indication: panel 39, turn on: valve 4, reading: control console 38
turn off: valve 4, reading: photodetectors 28
"spectroscopy
Xhorizontal > Xhoriz4
yes
delay
turn off: of all units, indication: panel 39
in accordance with the state of the console 38 turn on: valve 6
end

Fig. 4

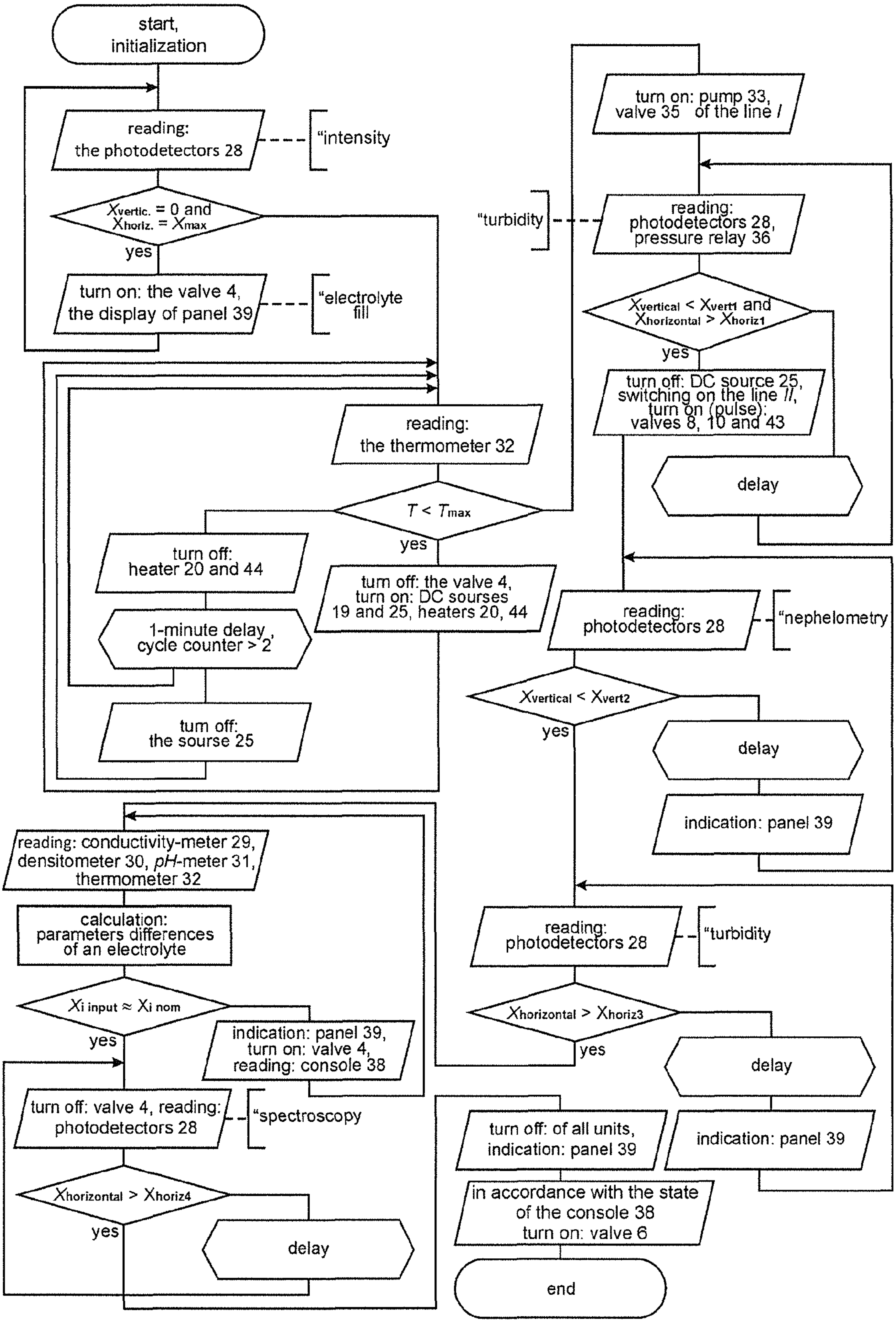
start, initialization
reading: the photodetectors 28
"intensity
Xvertic. = 0 and Xhoriz. = Xmax
yes
turn on: the valve 4, the display of panel 39
"electrolyte fill
reading: the thermometer 32
T < Tmax
yes
turn off: heater 20 and 44
1-minute delay, cycle counter > 2
turn off: the sourse 25
turn off: the valve 4, turn on: DC sourses 19 and 25, heaters 20, 44
turn on: pump 33, valve 35 of the line I
"turbidity
reading: photodetectors 28, pressure relay 36
Xvertical < Xvert1 and Xhorizontal > Xhoriz1
yes
turn off: DC source 25, switching on the line II, turn on (pulse): valves 8, 10 and 43
delay
reading: photodetectors 28
"nephelometry
Xvertical < Xvert2
yes
delay
indication: panel 39
reading: photodetectors 28
"turbidity
Xhorizontal > Xhoriz3
yes
delay
indication: panel 39
reading: conductivity-meter 29, densitometer 30, pH-meter 31, thermometer 32
calculation: parameters differences of an electrolyte
Xi input ≈ Xi nom
yes
indication: panel 39, turn on: valve 4, reading: console 38
turn off: valve 4, reading: photodetectors 28
"spectroscopy
Xhorizontal > Xhoriz4
yes
delay
turn off: of all units, indication: panel 39
in accordance with the state of the console 38 turn on: valve 6
end

Fig. 5

Europäisches Patentamt
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EUROPEAN SEARCH REPORT

Application Number
EP 15 20 0317

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X Y	US 2004/055964 A1 (KHALEMSKY ARON MIKHAILOVICH [RU] ET AL) 25 March 2004 (2004-03-25) * abstract * * figures 1, 5, 8 * * paragraphs [0002], [0021] - [0027], [0052] - [0058], [0064] - [0065], [0087] * -----	1,2 4	INV. C25D21/06 C25D21/18
X	WO 2008/144099 A1 (ITS ENGINEERED SYSTEMS INC [US]; SKINNER HARRY O [US]; GRIMME GREGORY) 27 November 2008 (2008-11-27) * abstract * * figures 1, 2 * * paragraphs [0004], [0019], [0034] * -----	1	
X	EP 0 560 175 A1 (HERBERTS & CO GMBH [DE]) 15 September 1993 (1993-09-15) * abstract * * figure 2 * * column 10, lines 6-37 * -----	1	**TECHNICAL FIELDS SEARCHED (IPC)**
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Place of search	Date of completion of the search	Examiner
The Hague	2 June 2016	Lange, Ronny

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